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NATIONAL BUREAU OF STANDARDS

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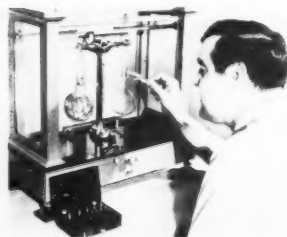
C. R. Smith, Secretary

NATIONAL BUREAU OF STANDARDS

A. V. Astin, Director

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COVER

To study air pollution, NBS developed several new analytical techniques. E. E. Hughes uses one of these techniques to determine the oxygen content of air by weighing the reaction products of the air and phosphorus. (See page 100.)

Prepared by the NBS Office of Technical Information and Publications, Washington, D.C. 20234

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The National Bureau of Standards serves as a focal point in the Federal Government for assuring maximum application of the physical and engineering sciences to the advancement of technology in industry and commerce. For this purpose, the Bureau is organized into three institutes—

- The Institute for Basic Standards
- The Institute for Materials Research
- The Institute for Applied Technology

The TECHNICAL NEWS BULLETIN is published to keep science and industry informed regarding the technical programs, accomplishments, and activities of all three institutes.

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INTERNATIONAL STUDY OF LASER WAVELENGTH

Lasers are being increasingly used in the field of metrology. One important application of gas lasers is their use as a wavelength source in interferometric devices for measuring length. In this instance it is necessary to know accurately the laser's wavelength as well as the reproducibility and long-range stability of this wavelength.

In a recent international undertaking, the same helium-neon laser was measured to determine its wavelength accurately and also to compare measuring processes. The study was conducted by K. D. Mielenz and K. F. Nefflen of the NBS Institute for Basic Standards, W. R. C. Rowley and D. C. Wilson of the National Physical Laboratory, and E. Engelhard of the German Physikalisch-Technische Bundesanstalt. Although each laboratory used a different procedure, none of the results differed from an average value by more than 5 parts in a billion. The study also showed that lasers, even of the same design, may emit different wavelengths and that wavelength may vary during the lifetime of the laser's discharge tube.

This round-robin measuring procedure, with the resulting close agreement, has thus clarified a discrepancy that appeared in 1965. At that time each laboratory measured a different laser of the same design and obtained results differing to a much larger degree.

At each laboratory, the wavelength

(nominally 633 nm) of the laser selected for international comparison was measured by comparing its wavelength to the international standard of length—the wavelength of the red-dish-orange light emitted by a krypton-86 lamp. In the NBS measurements, a polarization filter was used to match the light intensity of the laser to that of the krypton lamp. Both beams were then brought to a common focus with approximately the same angle of convergence at a small iris diaphragm. Next, the beams were recollimated to illuminate an interferometer with aluminized plates. The ring pattern produced by the interferometer was imaged on the

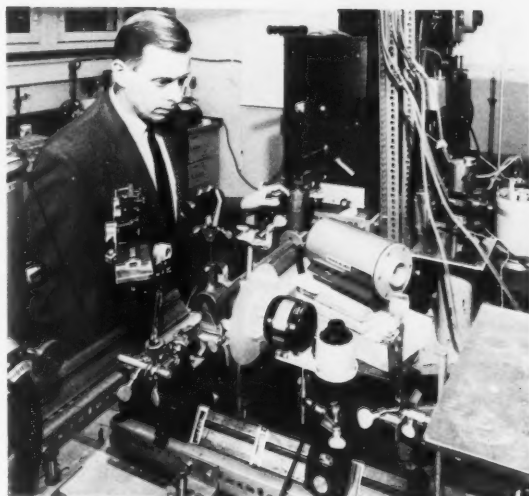
slit of a stigmatic plane-grating spectrograph.

The photographic plates containing the channeled spectra thus produced were evaluated with a photoelectric scanning microscope comparator. A computer program based on a least-square fit was used to calculate the laser wavelength relative to that of the primary standard line of Kr^{86} at 606 nm.

The result of the NBS measurements was a wavelength value of 632.991 420 nm. This value represents the mean of 10 observations. The measurements made at the National Physical Laboratory by a slightly different procedure resulted in a value

continued

K. F. Nefflen observes a fringe pattern in a spectrograph during a laser wavelength measurement.



K. D. Mielenz aligns the apparatus used to measure laser wavelengths. Light from a standard krypton-86 lamp (extreme left) is brought to a common focus with the laser beam (pointing toward reflector at right). The beams are recollimated to illuminate an interferometer (upper center) that produces a fringe pattern. This pattern is then photographically recorded by means of a spectrograph (right). Finally, a computer calculates the laser wavelength relative to the krypton-86 standard.

WAVELENGTH *continued*

of 632.991 416 nm, which is the mean of 20 observations. The average wavelength obtained at the Physikalisch-Technische Bundesanstalt was 632.991 418 nm.

The mean of the wavelength values obtained by the three laboratories was 632.991 418 nm; none of the three individual values differs from this value by more than 5 parts in 10^6 . Since three different and independent measurement techniques were involved in this intercomparison, it was concluded that there were no significant systematic errors in any of these techniques, and that 632.991 418 nm was the most likely wavelength of this particular laser.

In addition to the wavelength determination thus described, the three laboratories investigated a total of 13 other lasers in the study. It was found that an uncertainty of 1 part in 10^7 from the nominal value must be associated with any unmeasured laser wavelength. However, the measured wavelengths of secondary standards sources such as mercury-98 and cadmium-114 lamps have uncertainties of 5 to 8 parts in 10^6 . Therefore, although laser wavelengths can be determined somewhat less precisely than wavelengths from these secondary standards, they are sufficiently accurate for many industrial applications of laser interferometry.

On the other hand, the study has shown that laser wavelength is dependent on discharge-tube gas pressure and that the wavelength may vary during the lifetime of the laser's discharge tube. All of these findings point up the fact that, to obtain the highest accuracy, a laser used for length metrology should be calibrated and then remeasured from time to time during its lifetime. In view of these findings, NBS has initiated a program that will lead to a calibration service for lasers.

¹ For further details, see *Reproducibility of helium-neon laser wavelengths at 633 nm*, by K. D. Mielenz, K. F. Nefflen, W. R. C. Rowley, D. C. Wilson, and E. Engelhard, *Appl. Opt.* 7, No. 2, 289-293 (Feb. 1968).

PANORAMIC X-RAY MACHINE REDUCES COSTS

Study Shows Large Savings to the Government

■ A panoramic dental x-ray machine, developed at the National Bureau of Standards over a decade ago, was shown in a recent study to have saved the Government nearly \$1 million in the past fiscal year. The study was conducted by the machine's largest user, the Veterans Administration, who assessed the economic as well as other benefits of the device.

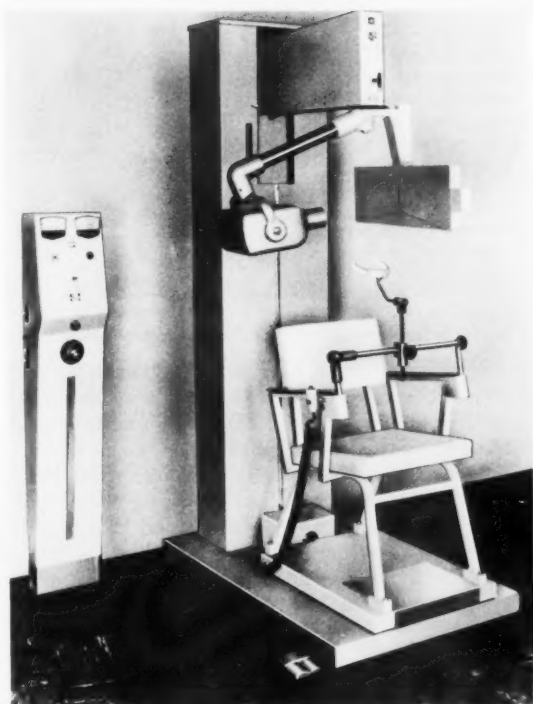
To date the machine has been used primarily for dental examinations of large groups of people because of the rapidity with which it can produce an x-ray picture of the entire dental arches. In contrast to slower conventional dental x-ray techniques, which require 14 to 20 individual exposures for a complete mouth record, the panoramic technique obtains a complete record with only one film.

The device was developed under Air Force sponsorship by J. W. Kumpula and G. Dickson, of the NBS dental research laboratory, and Dr. D. C. Hudson, then an NBS guest worker from the USAF Dental Service.¹ The Air Force, with its large number of inductees, was seeking a means to accelerate dental examinations of its military personnel. At present the machine is being used in a pilot study at a USAF inductee center and will be considered for further use at other bases.

Both the USAF and the VA have reported the saving of many professional and technical man-hours by use of the panoramic technique instead of conventional techniques. The VA study found that the time for a routine examination is reduced to approximately one-third. Additional savings are also effected through lower film costs and through reduced labor costs in handling one panoramic film compared to 14 to 20 individual films.

Increased Use Among Medical Specialists

Users of the panoramic x-ray machine are not limited to the field of dentistry however; medical specialists also



This commercial model of the NBS-USAF-developed panoramic dental x-ray machine is now in use by the Veterans Administration and the Air Force, as well as many civilian users.

A panorama of all dental structures is recorded on this 5 by 12 inch radiograph.

Although the center of the picture is unexposed (white area), no information is lost as the central incisors and associated areas are reproduced on both sides of the radiograph.



find it attractive for their needs. Particularly among eye, ear, nose, and throat specialists (as with a growing number of oral surgeons and orthodontists) the device is gaining wider use because more general diagnostic information can be obtained at a lower level of radiation than with conventional x-ray methods. Because the panoramic film reveals more of the tissue area and bony structure surrounding the dental arches, diseases and abnormalities in these regions are detected more frequently. At the same time, the absorbed dose of radiation is substantially reduced—7 to 13 times less² than with the multiple-film technique.

Design of the Panoramic Model

The basic design of the NBS-USAF prototype has been adopted in today's commercial model. With this device a narrow beam of x rays passes through the patient's head exposing an extraoral film on the opposite of the head. A panoramic x-ray picture of all the teeth and associated structures is recorded on a single 5 by 12 inch radiograph in 22 seconds.

In this machine, the x-ray source and film holder extend from an overhead vertical axis to either side of the patient's head. The source and film holder then follow semicircular paths on opposite sides of the head; after

completion of one semicircular path the chair shifts the patient to expose the other side of the head to the radiation beam. Rotation of the source (behind the patient) and the film (in front) is at such a rate that an x-ray shadow of each successive tooth falls on successive areas of the film. To avoid unwanted x-ray shadows from overlying bony structures, the narrow x-ray beam is made to enter the head largely through the soft tissue between the vertebral column and the bone of the jaw—first from one side, then from the other. And since the beam and film are in constant motion, shadows cast by intervening bone and other tissue, between the point of entry and the dental arch, move across the film too rapidly to obscure detail in the exposure.

The panoramic dental x-ray device, however, is not designed to supplant the conventional dental x-ray machine in cases where extreme sharpness and detail must be obtained. It is especially valuable in mass surveys where it will find use in child health and poverty programs.

¹A panoramic dental x-ray machine, by D. C. Hudson, J. W. Kumpula, and G. Dickson, U.S. Armed Forces Med. J. 8, 46 (Jan. 1957); NBS-AF panoramic x-ray machine, NBS Tech. News Bull. 40, No. 1, 1-3 (Jan. 1956).

²The integral absorbed dose in conventional and panoramic complete-mouth examinations, by J. Van Aken and L. W. Van der Linden, Oral Surg., Oral Med., Oral Pathol. 22, 603 (Nov. 1966).

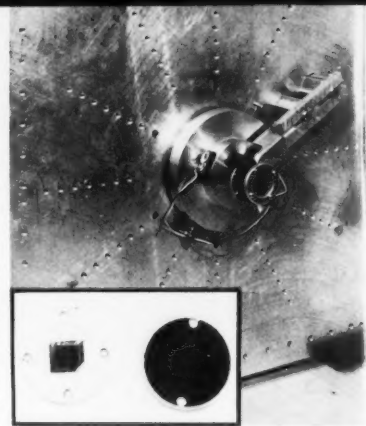
MÖSSBAUER EFFECT AIDS STUDIES OF GAGE BLOCK INSTABILITY

■ Mössbauer spectroscopy—based on the recoil-free emission and absorption of gamma rays—is proving a useful tool for determining the structure of metals. Recently, B. W. Christ and P. M. Giles of the NBS Institute for Materials Research explored the feasibility of applying this modern analytical technique to the study of dimensional instabilities in gage blocks.

Gradual changes in gage block dimensions are of great concern to today's machine tool and aerospace industries whose manufacturing operations demand reference standards of high precision and extremely small tolerances. Requirements may be foreseen for measurements to within 1 or 2×10^{-6} inch per inch of length, which require a gage block with a tolerance tenfold better, to within 1 or 2×10^{-7} inch per inch of length. Reference standards with this specified tolerance¹ have already been developed through Bureau research, together with measurement techniques² capable of calibrating to five parts in ten million, but anticipated requirements call for further improvements in both areas.

One way in which NBS scientists hope to achieve this objective is by a better understanding of the causes of dimensional instabilities. The reservoir of knowledge and

The steel foil absorber is shown in front of the velocity modulator, with the cobalt-57 source in position. Methods for supporting two types of samples (inset): a steel foil secured with cellulose tape, and an iron carbide powder (extracted from steel) dispersed in a plastic disk.



facilities available in the NBS metallurgy, metrology, and analytical chemistry laboratories makes a comprehensive approach to the problem possible. Phase transformations, as one cause of dimensional changes, are being examined by Dr. Christ and Mr. Giles using Mössbauer spectroscopy. With the Fe^{57} Mössbauer effect they have followed the progression of phase transformations and identified specific phases in metallurgical materials. Their initial results also show promise for quantitative analysis and for revealing details of atomic structure in such materials.³

Transitions Between Nuclear Levels

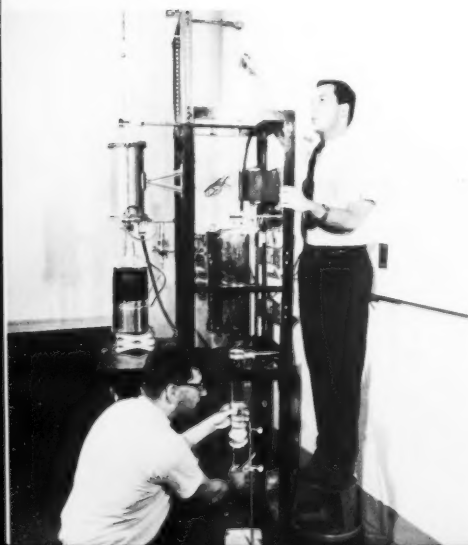
The Mössbauer effect—first reported by R. L. Mössbauer in 1958⁴—is based on a resonance phenomenon in which low-energy gamma rays, emitted from an isotope in the course of radioactive decay, are resonantly absorbed by another isotope having nearly the same characteristic frequency. Gamma ray emission occurs in the transition between nuclear energy levels, from the ground state to an excited state and vice versa. Mössbauer spectroscopy measures these transitions by embedding the isotope in a rigid crystal lattice to minimize its recoil. Therefore, because the isotope loses no energy through recoil, a true measure of the energies of the gamma rays is possible. The Mössbauer spectrum is obtained as a chart recording of the relative intensities of the varied energies of gamma rays emitted during transition.

In the NBS studies, transitions between nuclear energy levels were measured to learn more about the environment of the nucleus; that is, the metallurgical phase in which the nucleus is embedded and the local atomic arrangement near that nucleus. Such measurements can be made because nuclear energy levels vary with the electronic environment of the nucleus, which in turn depends on the kind and spatial arrangement of nearby atoms.

Thin Foil Samples

For Fe^{57} Mössbauer experiments any material containing natural iron is a suitable sample since 2.19 atomic percent of all natural iron is iron-57, an isotope that exhibits the Mössbauer effect. In this exploratory development work, experiments were conducted using specimens whose

Data were obtained on heat-treated samples. Treatment involved rapidly quenching a sample after it had been brought to phase equilibrium in the high-temperature, argon atmosphere furnace (center). Philip Giles (standing) will release the sample, suspended on a wire held with two magnets, as soon as a tube filled with ice water, held by Bruce Christ, is properly positioned.



structures were representative of those of complex gaging materials, yet simpler. Small thin foils ($1 \times 1 \times 0.004$ cm) of metallic iron and plain carbon steels were chosen to simulate the gage block structure, characteristically a low alloy steel (containing 1 weight percent carbon) which has been hardened by heat treatments.

Martensite-to-Carbide Transformation

From data obtained on these specimens, the NBS investigators demonstrated that characteristics of the Mössbauer spectrum such as line position and line width are sensitive to the carbon content and thermomechanical history of the specimens. Moreover, they found that the components of hypereutectoid steels hardened by heat treatment—high-carbon martensite and retained austenite—give rise to distinct lines in the Mössbauer pattern.

Of primary interest has been the martensite-to-carbide transformation, since it is the more predominant of two transformations occurring in heat-treated steels. The two components, high-carbon martensite and retained austenite, decompose at room temperature to form a carbide precipitate and more high-carbon martensite, respectively. During the course of a few years, this transformation results in a decrease in specific volume (the tetragonal lattice of martensite transforms to a more compact cubic lattice containing carbide precipitates) affecting the accuracy of the gage block.

Intermediate Structures Identified

In addition to following the progress of the martensite decomposition, a range of precipitate structures, previously unrecognized, has been detected and identified. When careful experimental procedures are followed, Mössbauer lines are observed for iron-carbon martensite, iron-carbon austenite, cementite (Fe_3C), and epsilon carbide. In the past a serious problem has been to detect small amounts of iron-carbon austenite, a constituent of steel that, when it spontaneously transforms to martensite, introduces undesirable properties such as brittleness.

Mössbauer Instrumentation

The principle of a Mössbauer measurement is quite simple,¹ although the instrumentation at times can be

most complex.² Essential elements are a radioactive source (nuclei in the excited state), an absorber (nuclei in the ground state) containing the same isotope, and a means of moving one relative to another. Gamma rays emitted at the radioactive source (cobalt-57 embedded in a cubic crystal lattice), when of nearly the same characteristic frequency as the sample isotope, are absorbed by the sample (iron-57 embedded in metallic iron). Any differences in their transition energies (due to differences in frequencies) are compensated for by the Doppler effect, through the use of a velocity modulator which moves the absorber relative to the source in the velocity range ± 1 cm/s. Thus, the energy at which resonance appears is derived by measuring the velocity that must be imparted to the absorber to restore resonance.

The data generated in this study are serving as essential background information for further thin foil measurements on the more complex structures characteristic of hardened gage block steels. Examination of bulk samples more typical of gage blocks and other metallurgical materials will depend upon successful development of a back-scattering spectrometer. At present, efforts are being made toward such development, both at the Bureau and elsewhere.

¹ Gage blocks of superior stability. II: Fully hardened steels, by M. R. Meyerson and W. A. Pennington, Trans. ASM 57, 3 (1964).

² Calibration of gage blocks, NBS Tech. News Bull. 45, No. 2, 26-30 (Feb. 1961).

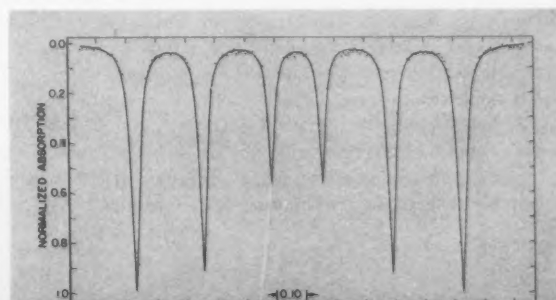
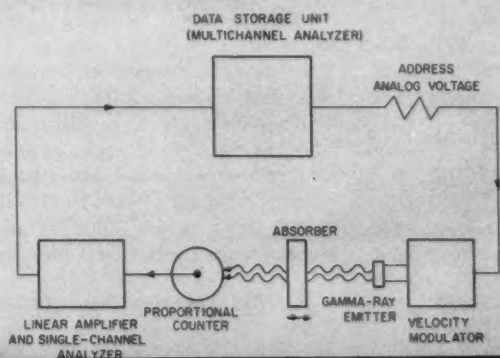
³ For further information, see Metallurgical uses of the Fe^{57} Mössbauer effect in metallic iron and plain carbon steels, by B. W. Christ and P. M. Giles, Advances in Mössbauer Methodology, Vol. 3, Plenum Press (in press); On the detection of retained austenite in high carbon steels by Fe^{57} Mössbauer spectroscopy (to be published).

⁴ Kernresonanzabsorption von gammastrahlung in Ir^{191} , by R. L. Mössbauer, Naturwiss. 45, 538 (1958).

⁵ Mössbauer effect, by G. K. Wertheim, Phys. Today 20, 31 (1967).

⁶ Constant acceleration Mössbauer spectrometer, by J. J. Spijkerman, R. C. Ruegg, and J. R. DeVoe, Rev. Sci. Instr. 36, 356 (1965).

Below left: Essential Mössbauer spectroscopy components include the velocity modulator, the moving absorber, and the proportional counter. The counter's output is amplified by the linear amplifier and passed to single-channel and multichannel analyzers, which store data and add repetitive scans. An address analog voltage provides synchronization between the multichannel analyzer and the velocity modulator. Below right: This Mössbauer spectrum of metallic iron, composed of six lines only 10^{-5} electron-volt apart reflects the resolution of the technique. The lines show the sudden increase in percent absorption for the isotope as a function of the absorber velocity.



NBS STUDIES ATMOSPHERIC GASES

Analytical Techniques Developed

■ Increased interest in the composition of the atmosphere, including normal components as well as accidental contaminants or pollutants, has provided the impetus for an intensive program in gas chemistry at the National Bureau of Standards. The program involves the development or improvement of gas analytical methods and the development and certification of gas standard reference materials. These standards are used as analytical controls and for calibrating gas measurement instrumentation and systems.

The gas chemistry program is being carried out in the Analytical Chemistry Division of the NBS Institute for Materials Research. Thus far it has provided analytical methods and standard reference materials for the determination of carbon dioxide and oxygen levels in air and oxygen levels in inert gases. Work on other atmospheric constituents is now in progress.

Carbon Dioxide Levels in the Atmosphere

Growing concern that atmospheric concentrations of carbon dioxide may be increasing, with a corresponding effect on both life processes and climatic conditions, has stimulated interest in measuring the carbon dioxide level of the atmosphere. Unfortunately, this level is normally so low—approximately 340 ppm—that classical absolute methods are not very practical. Such procedures are either volumetric, involving the measurement of the volume change of the gas after absorption of carbon dioxide in a suitable reagent, or gravimetric, fol-

lowing absorption on a solid alkaline reagent. These techniques are time consuming and require a considerable degree of skill on the part of the analyst.

Instrumental methods are more convenient but require accurately certified standard reference materials for their use. To meet this need, NBS has recently developed three gaseous standard reference materials consisting of nitrogen containing carbon dioxide at 308, 346, and 384 ppm, respectively, and hence bracketing the atmospheric level.¹ These mixtures were prepared by quantitative dilution of more concentrated mixtures. After each dilution step, the mixtures were analyzed by absolute measurements.

In the course of this work, an improved method for the determination of carbon dioxide by mass spectrometry was developed by E. E. Hughes and W. D. Dorko.² Conventional mass spectrometry is not sufficiently sensitive to permit determination of CO₂ at the 340 ppm level with the required precision of one relative percent. In the improved method, the equipment is operated at increased sample pressure, thereby enhancing the signal to permit this precision. To obtain valid results, the equipment must be calibrated at the increased pressure. The use of the NBS carbon dioxide-in-nitrogen standards provides the necessary calibration.

Oxygen Content of Air

The determination of oxygen at atmospheric levels is accomplished classically by absorption of oxygen in

a reagent such as alkaline pyrogallol or cuprous chloride with measurement of the corresponding volume change. The difficulty of this procedure is such that oxygen determinations accurate to 0.01 percent of oxygen are seldom undertaken.

In the production of a "standard air" with a certified oxygen content³ as a reference material for instrumental techniques, Mr. Hughes developed an absolute method for oxygen determination that is both accurate and simple.⁴ In this method, air is admitted to an evacuated flask containing either white phosphorus or a sodium-potassium alloy. The flask is weighed, after which the unreacted gases, nitrogen and argon, are pumped away. A reweighing permits the determination of oxygen in the original sample which chemically combined with the phosphorus or alloy to form an oxide.

With this technique, the oxygen composition of air near the NBS Gaithersburg site in rural Maryland was found to be 20.95 ± 0.02 mole percent. This value is in close agreement with a value found by Benedict in 1912⁵ for the oxygen content of air in the vicinity of Washington, D.C., indicating that probably no significant change has occurred in the overall oxygen content of the atmosphere during the elapsed interval.

Low Level Oxygen Concentrations

The determination of oxygen at levels other than atmospheric is often of interest. For example, oxygen must be excluded in many situations, and determining its very low amounts in so-called inert blanketing gases is important. Another example is the considerable interest in determining the intermediate levels of this gas in respired air.

To provide for the calibration of analytical instrumentation for determining low oxygen concentrations, NBS has developed a series of standard reference gases with specific oxygen contents of 0.0978, 0.0212, 0.0112, 0.00103, and 0.000303 mole percent, respectively.⁶ To certify these

gases, an absolute electrochemical method of analysis was worked out by J. M. Ives, E. E. Hughes, and J. K. Taylor.⁶ In this method, the gas is passed through a galvanic cell with silver and lead electrodes that are covered with a film of potassium hydroxide solution. The current output of this cell depends on the rate of oxygen consumption, which in turn is related to oxygen content and rate of flow of the gas, as well as to certain kinetic parameters.

Because of the kinetic parameters, the cell is not 100 percent efficient; hence, the laws of electrolysis cannot be applied as in coulometric procedures. To overcome this problem, the gas is passed successively through several cells so that the oxygen is essentially completely reacted. Moreover, the measurement is conducted at two different rates of flow to cancel out the kinetic effect. The difference

in total current for the two experiments can then be equated to the absolute oxygen content by use of quantitative electrolytic considerations.

This method is very simple to use, yet it provides analyses that are accurate to at least 1 part in 100 in the concentration range between 0.1 and 0.0001 mole percent of oxygen.

Galvanic cells similar to those of the NBS technique have been known for some time but have been used empirically in the single-cell mode of operation because of an imprecise knowledge of the kinetic factor already mentioned. The NBS-developed arrangement offers some convenience of use and minimization of sample requirement, while at the same time removing the need for calibration. With the new standards, however, galvanic cells may be calibrated and thus used in a single-cell mode of operation for comparative measurements.

In addition to the activities already described, the gas analysis group plans to investigate analytical methods and provide standards for certain atmospheric pollutants such as sulfur dioxide, carbon monoxide, and hydrocarbon gases. The results of this program will be described as the work progresses.

¹ NBS Standard Reference Material Nos. 1601, 1602, 1603. See, NBS Tech. News Bull. 51, No. 8, 180 (Aug. 1967).

² Accurate mass spectrometric determination of low concentrations of carbon dioxide in nitrogen, by E. E. Hughes and W. D. Dorko, Anal. Chem. 40, 750-755 (1968).

³ See, Standard Reference Materials, page 102 of this issue.

⁴ A simple technique for the determination of atmospheric oxygen, by E. E. Hughes, Environ. Sci. Technol. 2, No. 3, 201-203 (1968).

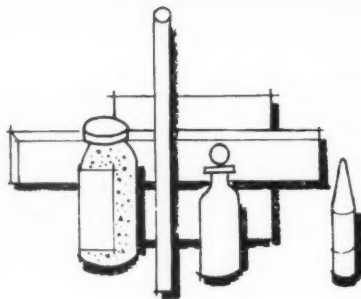
⁵ The Composition of the Atmosphere with Special Reference to Its Oxygen Content, by Francis G. Benedict, Carnegie Institution, Washington, D.C., p. 114 (1912).

⁶ Absolute determination of low concentrations of oxygen in inert gases using galvanic cells, by J. M. Ives, E. E. Hughes, and J. K. Taylor (in preparation).



Julian Ives determines trace amounts of oxygen in gaseous mixtures. The oxygen is essentially completely reacted in the four cells (center) and produces an electric current which is equated to the oxygen content of the gas.

STANDARD REFERENCE MATERIALS



Standard Reference Materials are well-characterized materials disseminated by NBS to be used in calibrating and evaluating measuring instruments, methods, and systems or to produce scientific data that can be referred readily to a common base. These materials are certified for chemical composition or for a particular physical or chemical property. They are used on-site in science and industry for calibrating the instruments and methods used for production and quality control of raw materials, chemicals, metals, ceramics, fuels, and radioactive nuclides in manufacturing processes and in research. This column regularly reports on the issuance of new and renewal Standard Reference Materials and on latest developments in the Standard Reference Materials Program.

Oxygen-in-Nitrogen Standards

The NBS Office of Standard Reference Materials has prepared a new series of gaseous standards containing concentrations of oxygen in nitrogen over the range from about 1 part per million of oxygen up to the level of oxygen in the atmosphere.¹

The new standards are supplied in units of 68 liters at a pressure of 500 psi in disposable gas cylinders. The identification numbers and the normal oxygen concentration levels are:

| NBS No. | Oxygen Content (mole percent) * |
|---------|---------------------------------|
| 1604 | 0.000 303 ± 0.000 001 |
| 1605 | 0.001 03 ± 0.000 01 |
| 1606 | 0.0112 ± 0.0001 |
| 1607 | 0.0212 ± 0.0002 |
| 1608 | 0.0978 ± 0.0007 |
| 1609 | 20.95 ± 0.02 |

The price of each standard is \$105 per unit (cylinder).²

The preparation and certification of the oxygen-in-

*The uncertainty shown represents the 95 percent confidence limit of the mean based on 20 determinations of the oxygen content of six samples selected at random from a total lot of 20.

nitrogen standards required the development of new and modified analytical methods³ for determining oxygen in gases. These methods made possible the certification of the oxygen contents of the standards to one relative percent or better at all concentration levels.

The oxygen-in-nitrogen standards are intended primarily for calibrating instruments used to detect the presence of oxygen and those used to determine the oxygen concentration in a gaseous mixture. In metallurgy, in the aerospace industry, and in the processing of solid state electronic components, blanketing gases are used to displace oxygen during critical operations. Such use is increasing and requires the monitoring of low concentrations of oxygen. At intermediate levels of oxygen content, standards for calibration of gas chromatographs and other instruments for monitoring oxygen concentrations are required. Standards containing oxygen at atmospheric concentrations and those approximating the oxygen content of respired air are also critically needed. For example, in medical research there is a great need for primary standard reference gases in the study of pulmonary and related diseases.

Until now, no basic standards existed which could be used to calibrate or unequivocally intercompare various instrumental techniques for the determination of oxygen at all levels of concentration. This series of oxygen-in-nitrogen standard reference materials, by virtue of the care with which they have been compounded and analyzed, will provide basic reference points for users in many areas of science, industry, and medicine. They complement the carbon dioxide-in-nitrogen standards (NBS Nos. 1601, 1602, and 1603, which cost \$148 per unit) issued last year.²

Standard Metallo-Organic Compounds

Three additional metallo-organic standards for determining metals in petroleum products were recently re-

newed¹ and certified. They are: NBS Nos. 1077a, Silver 2-Ethylhexanoate; 1051b, Barium Cyclohexanecarboxylate (IV); and 1052b, Bis(1-phenyl-1,3-butanedione)oxovanadium. These standards were prepared to fill the need for an adequate collection of standards that could be used to prepare a desired blend of known metal concentration in an appropriate lubricating oil.

The standards are supplied in units of approximately 5 grams at a price of \$26 per unit.² The certificate furnished with each standard gives the amount of the certified metallic element present, as well as directions for preparing a solution of known concentration in lubricating oil.¹

The amount of wear on components of an internal combustion engine may be determined by analyzing its lubricating oil for metals. Such analysis is done with the optical emission spectrometer. Accurate predictions of wear or probable failure ordinarily are made on the basis of trends in data accumulated over a period of time from a series of determinations. To assure the accuracy of the analytical work, the spectrometer must be calibrated with standard reference materials.

The Bureau has developed a series of 24 different

metallo-organic standards for this purpose at the request of the Division of Refining of the American Petroleum Institute. These standards are stable, are oil-soluble at the concentrations needed, and do not absorb excessive amounts of water. They yield solutions in lubricating oils which are constant and which do not precipitate on standing.

The principal users of the metallo-organic standards were at first the railroad and trucking industries. The analysis for metals in lubricating oils was used as the basis for maintenance schedules and predictions of equipment failure. Agencies of the Department of Defense also utilize the metallo-organics for safeguarding equipment and for minimizing maintenance and repair costs. Newer uses include monitoring the presence of catalyst metals and catalyst poisons in process streams. The increasing uses for the metallo-organic standard reference materials and needs for additional metallic elements in the compounds are expected to require an increase in the scope of the metallo-organic compound program.

These metallo-organic compounds were prepared by Distillation Products Industries, Rochester, N.Y. Chemical analyses were made by B. B. Bendigo, R. A. Durst, and R. A. Paulson, and spectrochemical analyses by Virginia C. Stewart, all of the Analytical Chemistry Division, NBS Institute for Materials Research.

Correction

On page 41 of the February 1968 issue of the Technical News Bulletin, this column reported on Gas-in-Metal Standards. Unfortunately, there was an error in the fourth paragraph dealing with the determination of activation values. That paragraph reads in part, "... by using the n, gamma reaction." It should have read, "... by using the (n, p) reaction."

¹ For a complete list of Standard Reference Materials available from NBS, see *Standard Reference Materials: Catalog and Price List of Standard Materials Issued by the National Bureau of Standards*, NBS Misc. Publ. 260 (1968 ed.), for sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for 45 cents. Quarterly insert sheets which update Misc. Publ. 260 are supplied to users on request.

² These standards may be purchased for the price indicated from the Office of Standard Reference Materials, Rm. B308, Chemistry Bldg., National Bureau of Standards, Washington, D.C. 20234.

³ (a) Accurate mass spectrometric determination of low concentrations of carbon dioxide in nitrogen, by E. E. Hughes and W. D. Dorko, *Anal. Chem.* **40**, No. 4, 750-755 (Apr. 1968).

(b) A simple technique for the determination of atmospheric oxygen, by E. E. Hughes, *Environ. Sci. Technol.* **2**, No. 3, 201-203 (Mar. 1968).

(c) Absolute determination of low concentrations of oxygen in inert gases using galvanic cells, by J. M. Ives, E. E. Hughes, and J. K. Taylor (in preparation).

(d) See also, NBS studies atmospheric gases, page 100 of this issue.

⁴ *Analytical Standards for Trace Elements in Petroleum Products*, NBS Mono. 54 (1962). Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for 25 cents.

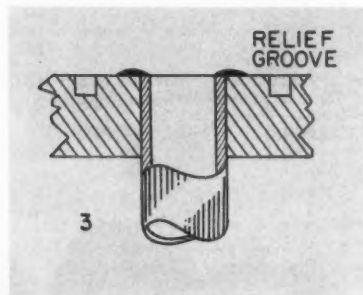
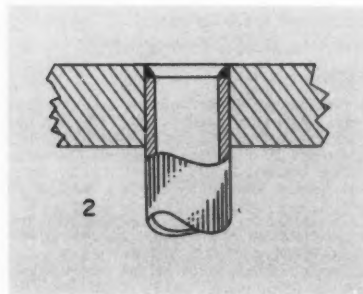
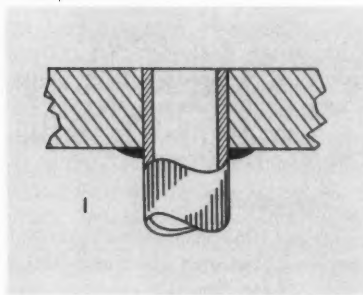


Cylinders of a standard reference gas are filled from the Bureau's master mixture of the gas.



Stainless steel welded to stainless steel illustrates the very smooth surfaces that can be attained with the "corner-to-corner" welding technique.

WELDING TECHNIQUE FOR HIGH-VACUUM EQUIPMENT



■ To meet the needs of NBS scientists for equipment that can withstand high and ultrahigh vacuum, Ralph Orwick of the NBS instrument shops has developed a welding technique that eliminates many of the drawbacks associated with earlier welding methods. With this technique, very close tolerances can be attained and the very smooth surface that results eliminates costly post-welding machining. This method has also been successfully used to weld heretofore "unweldable" combinations of metals, such as stainless steel to steel, stainless steel, inconel, monel, and kovar; inconel to inconel; monel to monel; and titanium to titanium.

To illustrate how the new technique avoids the disadvantages of the earlier methods, consider the problem of welding a tube to a flange. In the original method, the tube was inserted into a hole in the flange and welded externally. The disadvantage in this method was the internal crack left between the tube and the sides of the hole, which had to be evacuated (Fig. 1). The solution to the problem was internal welding—the second method (Fig. 2). Unfortunately, this method, though providing a better vacuum, caused the flange to warp. To eliminate the warping, a relief groove surrounding the hole was cut in the flange (Fig. 3). This method of weld-

ing, with some variations, is the one most widely used today.

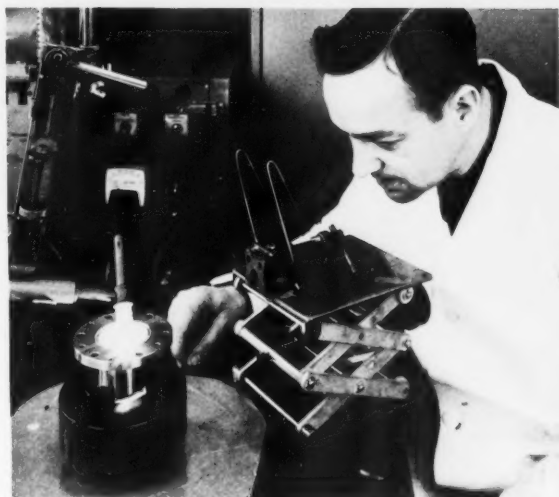
The NBS technique also uses an internal weld, but the need for relief grooves and post-welding machining has, for the most part, been eliminated. This method has two main advantages: it results in a very smooth surface that leaves no crevice in the metal to be evacuated, and it may be used where space limitations prohibit the machining of relief grooves. Also, the flange rests on a shoulder, which reduces the stress on the weld and thus reduces the chance of the weld's failing.

In the NBS technique, two concentric holes are drilled into the flange. The smaller penetrates the flange while the larger stops 0.030 in before penetration is achieved. This leaves a shoulder in the hole. The diameter of the smaller hole is 0.060 in larger than the inside diameter of the tube to be joined to the flange, while the larger hole matches the outside diameter of the tube.

If the shoulder is properly machined, the machined end of the tube will rest flush against it. As such machining may prove difficult, particularly in the corner, the outside diameter of the tube end should be beveled slightly to ensure that the tube seats properly.

When the tube and the flange are

CHNIQUE



Above: Ralph Orwick uses the "corner-to-corner" welding technique.

Right: The welding apparatus is positioned so that the tungsten electrode forms a 45° angle with the vertical and is 0.010 to 0.015 in away from the junction of the two parts being welded.



joined, the shoulder and tube end form a ledge 0.030 in deep and 0.030 in wide (Fig. 4). The welding torch is directed at the junction of the tube and shoulder, causing the metal of both parts to flow toward the junction and form a "corner-to-corner" weld.

Material Preparation

As in any welding operation, the quality of the weld depends greatly on the preparation of the materials. The parts must be properly machined and cleaned. The weld area must be free of all burrs that would trap oil and dirt and prevent thorough cleaning.

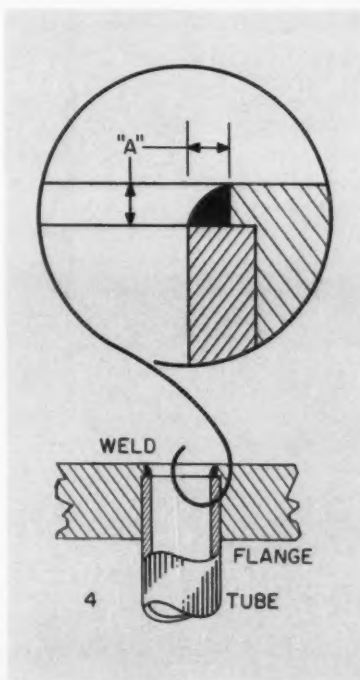
The parts should be cleaned by a hot vapor degreaser of trichloroethylene. While still hot, they should be washed in acetone. The acetone removes any residue of trichloroethylene which would "outgas" in a vacuum system. If a hot degreaser is not available, some other cleaning

agent may be used—such as freon degreaser, which is available in spray cans.

Welding Procedure

After the flange and tube have been machined and cleaned, they should be assembled and "tack-welded" in two or more places before starting to weld. The assembly is positioned with the flange up and in the horizontal plane. The welding apparatus is placed so that the tungsten electrode forms a 45° angle with the vertical and is 0.010 to 0.015 in away from the junction of the flange shoulder and the tube end. The electrode tip should be ground to a point to give better control of the arc. Careful control of the arc temperature prevents build-up of weld metal; the edge of the flange hole and the end of the tube flow toward their junction, producing a smooth, even convex weld. To assure such a weld, the electrode is held stationary and the assembly

continued



WELDING TECHNIQUE *continued*

is rotated about the tube axis by a motor-driven positioner.

To eliminate oxidation, welding is performed in an inert gas atmosphere consisting of 60 percent helium and 40 percent argon. This gas mixture flows about the electrode at the rate of 10 cubic feet per hour. What little oxide does form can easily be removed by brushing the weld lightly, while still warm, with a rotary-drive, stainless-steel brush.

No filler wire is added to the welded joint. Without a filler wire, the required temperature is less, and the chance of producing a porous weld is reduced. A porous weld may not necessarily leak, but attaining an ultra-high vacuum with it is difficult, if not impossible.

This method does not necessarily produce a strong weld; therefore, if any strain is to be applied to the flange, the flange should be tack-welded on the outside for strength. However, the outside weld should not be solid; at least two or more spaces should be left in it to permit checking of the inside weld for leaks.

The NBS method has generally been used with $\frac{1}{16}$ in thick wall tubing, although thicknesses from

0.003 to $\frac{1}{8}$ in and more have been used. Several variations of this technique can be used depending on the particular application.

For a wall thickness of less than 0.015 in, a heat sink must be used to prevent warping and a retainer ring must be used to ensure that the tubing does not pull away from the flange during welding.

The NBS method can be used when the flange is inside the tube. However, with thin tubing it is again necessary to use a heat sink while welding. This is done by matching a ring (preferably copper) that fits the outside diameter of the tubing.

There are several ways to adapt this technique to thick-walled tubing. First of all, a heavier weld is needed. For wall thicknesses greater than $\frac{1}{16}$ in, 0.045 by 0.045 in corners are suggested; for welds larger than 0.060 in, a filler wire should be used.

When a hole already exists in the flange and it is not possible to turn a shoulder for the tubing, a corner-to-corner weld can be produced by thinning the tubing's inside diameter to the desired thickness. It need only be thinned back a short distance (approximately $\frac{1}{16}$ in) on the end being welded. When the tube is inserted into the flange it should be aligned so that the tube is below the surface of the flange a distance equal to the thickness of the thinned tube section. An added precaution is to recess, for a short distance, the area around the hole by approximately $\frac{1}{16}$ in below the flange surface. This will tend to eliminate the need to machine the weld if it should build up higher than the recessed surface.

Another application for this technique is in welding a small tube to the side of a larger tube. Here it is necessary to match the end of the small tubing to the curvature of the larger tubing's inside diameter. The larger tubing should have a thicker wall than the smaller. The thinnest wall that is recommended for the larger tubing in this application is $\frac{1}{16}$ in.

If a thicker than 0.030 in wall is being used for the smaller tubing, the

end of the tubing being welded should be bored or turned to 0.030 in wall thickness.

The small tubing is inserted into a hole machined in the side of the large tube. The outside diameter of the small tube fits fairly tightly in the hole. The small tube is inserted until it is even with the inside wall of the large tube. For corner-to-corner weld preparation, the small tube is pulled back 0.030 in from the inside wall of the large tube.

All joint preparations mentioned thus far have been those that can be reached from the inside of the vacuum system. However, it is not always possible to design every joint of a vacuum system where it can be welded on the inside.

In such cases the full melt-through method is used. In this method, the pieces, or system to be welded together, are first backfilled with an inert gas (usually argon). The gas is introduced into one end or port of the system, displacing all the oxygen. The system is closed with the exception of one small opening, which allows the gas to escape, thus preventing pressure buildup during welding. Enough amperage is used to melt through both pieces being joined together. The result is a smooth oxide-free weld on the inside. Any oxide that forms on the outside can be removed by brushing with a wire brush while it is still warm. To prevent oxidation after welding, the inert gas flow should be continued until the welded piece is cool.

The full melt-through method is used to join two pieces of tubing of the same material having the same inside and outside diameters. Heavy wall tubing (over 0.083 in) should be beveled on the outside and a filler rod used to ensure full penetration. This method is also used to join a tubing to a blank flange or one where the hole in the flange is smaller than the inside diameter of the tubing. This is done by machining on the flange a mating lip that has the same inside and outside diameters as those of the tubing.

Titanium was welded to titanium to form a part for use in the Bureau's linear accelerator (LINAC). (Weld—arrow.)





STANDARDS AND CALIBRATION

AWARDS RECOGNIZE WWV CONTRIBUTIONS

Transmissions from the Bureau's radio station WWV are used as standards not only by industry, governments, and educational institutions, but also by amateur radio operators. WWV's contributions to amateur radio have now been recognized by the International Amateur Radio Union (IARU) and the American Radio Relay League (ARRL). IARU awarded its "Worked All Continents" (WAC) certificate and ARRL its "Worked All States" (WAS) certificate. They normally are given on the basis of two-way communications; their award to WWV is unusual in that it is for one-way communications.

Amateur radio operators—"hams"—rely on radio signals from WWV to calibrate their receivers, and thereby accurately determine their transmitter frequencies, also. This is done by setting a calibration oscillator so that its appropriate harmonic matches the WWV frequency being received, and then using the harmonics as calibration points. Many hams use this technique to calibrate their receivers at all received multiples of 100 kHz.

The ready availability of WWV signals for standards has greater significance than enabling operators to avoid the penalties of transmitting on unauthorized frequencies. It has also contributed to the orderly use and maximum utilization of the radiofrequency spectrum.

STANDARD FREQUENCY AND TIME BROADCASTS

WWV—2.5, 5.0, 10.0, 15.0, 20.0, and 25.0 MHz

WWVH—2.5, 5.0, 10.0, and 15.0 MHz

WWVB—60 kHz

Radio stations WWV (Fort Collins, Colo.) and WWVH (Maui, Hawaii) broadcast signals that are kept in close agreement with the UT2 scale by making step adjustments of 100 ms as necessary. Each pulse indicates that the earth has rotated approximately 15 arcseconds about its axis since the previous one. Adjustments are made at 0000 UT on the first day of a month. *There will be no adjustment made on June 1, 1968.* The pulses occur at intervals that are longer than one second by 300 parts in 10^{11} due to an offset in carrier frequency coordinated by the Bureau International de l'Heure (BIH), Paris, France.

Radio station WWVB (Fort Collins, Colo.) broadcasts seconds pulses derived from the NBS Time Standard (NBS-III) with no offset. Step adjustments of 200 ms are made at 0000 UT on the first day of a month when necessary. BIH announces when such adjustments should be made in the scale to maintain the seconds pulses within about 100 ms of UT2. *There will be no adjustment made on June 1, 1968.*

David Andrews (right), recently retired chief of NBS Radio Broadcast Services, hangs the WAC award for Peter Vitezic, Jr. (left), his successor, while Leo Honea, engineer-in-charge of WWV, positions the WAS award. In the background are some of the QSL cards—verifications of reception—sent to WWV by hams of many countries.



DATA OBTAINED ON DIELECTRIC CONSTANT AND LOSS FACTOR OF FOLIAGE

■ Data on the dielectric constant and dissipation factor of foliage were recently obtained by M. G. Broadhurst of the NBS Institute for Materials Research. In this work, which was sponsored by the Navy, live plant materials were measured for their dielectric properties at various moisture contents and temperatures. The data are expected to be useful in improving the design of radio antennas for use in jungles and other heavily wooded areas.

In the NBS study, values of dielectric constants of the materials up to frequencies of about 4×10^9 Hz were obtained from measurements of the admittance of a coaxial cylindrical transmission line with a specimen of the material occupying some of the space between the coaxial conductors. The admittance of the part of the line occupied by the sample was shown to be the capacitive susceptance of the empty line enhanced by the relative complex dielectric constant.

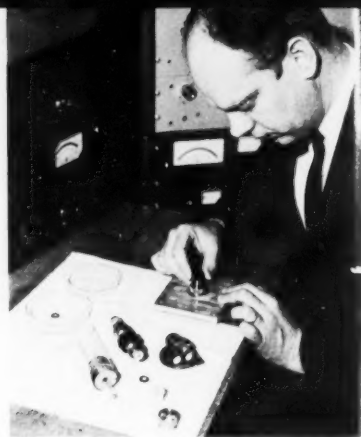
Measurements of the coaxial line admittance at frequencies from 0.1 MHz to 250 MHz were made with a bridge to determine the capacitance and conductance of the coaxial line. Measurements at higher frequencies were made with a slotted-line technique.

Because a leaf has a high dielectric constant (similar to that of water), the effect of an air gap between the sample and holder is severe. That is, the sample can be considered a large admittance in series with a comparable size gap admittance. The air-gap errors were reduced by measuring the leaves immersed in water, thereby increasing the gap admittance by a factor on the order of 100 and also preventing drying of the leaves during measurement.

The leaf samples were cut to fit snugly in a sample holder by means of a specially made "cookie cutter." Immediately after cutting, the sample was weighed, measured for thickness, and immersed in distilled water. With the sample holder also immersed and all visible bubbles removed, the leaf wafer was placed in the sample pocket and a cap secured. The unit was then taken to the various measurement systems for electrical measurements. The first measurement was always repeated at the end of a run to see what electrical changes, if any, had occurred.

Moisture content was determined by weighing the specimen before measurement and after completely drying it

M. G. Broadhurst cuts leaf wafers for a study of the dielectric properties of foliage.

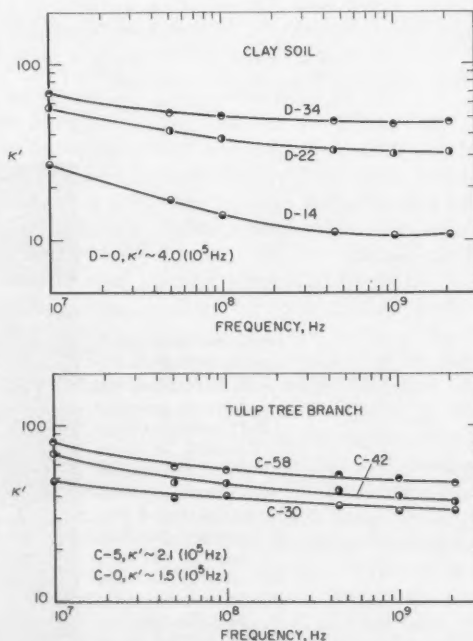


in a warm oven. The weight loss divided by the initial weight was taken to be the proportional moisture content of the leaf during measurement.

The measuring system was checked for accuracy by measuring the empty holder with the sample chamber containing air. Distilled water was then placed in the chamber and the admittance measurements repeated. The dielectric constants of water thus obtained were within 10 percent of the known values.

The specimens were collected locally from living plants, and measurements were made shortly after removal from the plants. The specimens included leaves from bamboo,

continued on page 115



Plots of data for the three samples of tulip tree branches and three samples of clay soil as a function of frequency at 23°C. The sample number denotes the moisture content (weight percent) of the sample. At the lower left of each graph is the value for a dry sample.

ESTIMATING MOLECULAR WEIGHTS OF VINYL ACETATE COPOLYMERS IN LATEX PAINTS

A rapid procedure for estimating the apparent molecular weights of vinyl acetate copolymers in latex paints has been developed at NBS. The work of Harvey W. Berger, this procedure involving viscometric measurements can readily be used by industrial and governmental coatings laboratories to characterize paint performance on the basis of molecular weight.

Several of the important properties of a latex paint depend upon the molecular weight of the polymer used in it: these properties are improved when high molecular weight polymers are used. Such properties include durability, scrubability, hardness, flexibility, and resistance to abrasion, heat, solvents, and water.

Various procedures, such as osmotic-pressure and light-scattering techniques, are used to determine the molecular weights of polymers. Most of these techniques have several drawbacks when they are applied to methods involving paint. Some require delicate or expensive equipment; others are adversely affected by the fine pigment particles that cannot be completely extracted from the paints. These disadvantages and the need for a rapid and precise method of analysis have led to consideration of viscometry as a suitable alternative.

Viscosity measurements of dilute solutions have been shown to be relatively insensitive to interferences caused by pigments or by such paint additives as thickeners, wetting agents, and fungicides. The apparatus required for such measurements is simple and inexpensive.

Although the viscosities of polymers are closely related to their molecular weights, the measurement of viscosity does not lead directly to values for absolute molecular weights. In the NBS method an empirically determined logarithmic equation relating average apparent molecular weights, \bar{M} , to intrinsic viscosity, $[\eta]$, is used to estimate the molecular weight of the vinyl acetate copolymers. This equation takes the form: $\log \bar{M} = 1.45 \log [\eta] + 5.521$. The procedure provides sufficient accuracy to obtain measurements with an uncertainty of 2000 in the 200 000 to 75 000 molecular weight ranges.

The equation was derived from the Mark-Houwink equation, $[\eta] = KM^a$, in which the intrinsic viscosity, $[\eta]$, is a limiting factor obtained graphically by extrapolating from experimental measurements of specific viscosity. The values of parameters a and K depend on the type of polymer and solvent being considered, as well as other structural properties of the polymer.

There are theoretical limitations to the use of the Mark-Houwink equation in terms of the extent of the non-linearity and cross-linking of the polymer, and generalizations involved in the use of the literature values for a and

K . These difficulties are not serious when applied to polymers of broad, undefined properties.

Literature data for determinations of molecular weights of polyvinyl acetate copolymers in acetone were reviewed, and an empirical evaluation was used to derive values for a and K which apply to a broad range of molecular weights.¹ The a and K values were substituted in the equation, $[\eta] = 1.55 \times 10^{-4} \bar{M}^{0.69}$, which, expressed logarithmically and rearranged, gives

$$\log \bar{M} = 1.45 [\eta] + 5.521.$$

Experimental Procedure

Seven latex emulsion samples of various copolymers were used to formulate seven experimental paints in the laboratory. Intrinsic viscosities for the polymers in both the latex emulsions and the whole paints were obtained and compared. In addition, the molecular weights of the polymers in six commercially available paints were determined to ascertain the molecular weight range of materials used in such commercial products.

The whole paint samples, which were calculated to contain approximately 0.5 g polymer, were digested with boiling acetone. Pigments and other acetone-insoluble materials were separated by centrifuging and the solution of polymer in acetone was decanted into volumetric flasks and diluted with acetone. The latex emulsion samples were also calculated to contain approximately 0.5 g polymer and were similarly dissolved in acetone and diluted. The weight of polymer in each sample was then determined by evaporating the acetone solution and weighing the residual polymer.

Viscometer flow times were measured for each of the polymer solutions and for three dilutions of each solution. Viscosity measurements were made using a liquid flow viscometer for liquids in the 0.4×10^{-6} to 1.6×10^{-6} m²/s (0.4 to 1.6 centistoke) range, giving flow times in excess of 250 seconds. The viscometer and its contents were maintained at $25^\circ\text{C} \pm 0.5^\circ\text{C}$ in a constant temperature bath and flow times were measured with a stopwatch readable to within 0.1 second. From these data, the specific viscosity, η_{sp} , for each concentration was calculated. The intrinsic viscosity was then obtained graphically by plotting specific viscosity divided by concentration, C , against concentration of the polymer in acetone. The intrinsic viscosity was defined as the value of η_{sp}/C when C equals zero.

¹ The estimation of molecular weights of vinyl acetate copolymers in latex paints, by H. W. Berger, *J. Paint Technol.* **39**, No. 508, 310-315 (May 1967).

CONFERENCE & PUBLICATION *Briefs*

ELEVENTH SCINTILLATION AND SEMICONDUCTOR COUNTER SYMPOSIUM

More than 500 scientists from over 10 countries gathered in Washington, D.C., from February 28 to March 1, 1968, to attend the 11th Scintillation and Semiconductor Counter Symposium. The Symposium presented the latest concepts and developments in nuclear radiation detection.

Interest in nuclear detection has grown rapidly over the years as evidenced by increasing attendance at the symposia. The first of the series of meetings of this kind was held at the University of Rochester in 1948 and was attended by about 40 persons from this country. The latest symposium in the series was attended by about 550 persons representing a worldwide audience.

These meetings are jointly sponsored by the Nuclear Science Group of the Institute of Electrical and Electronics Engineers, the National Bureau of Standards, and the Atomic Energy Commission. G. A. Morton of RCA (Princeton) served as general chairman of the Symposium as he has for a number of years.

One of the highlights of the meeting was a banquet speech on the first evening given by W. N. Hess, Director of Science and Application at the Manned Spacecraft Center, Houston, Tex. Dr. Hess detailed the aims of the moon space programs. Our mission, he explained, is not just to see if we can get there, but to try to bring back and analyze the material found. Scientists hope to learn about the properties and quantities of materials found on the moon. In addition, long-range tests will be performed to analyze the magnetic fields and seismic disturbances using instruments left on the lunar surface to transmit signals back to earth. The speaker also discussed both the material and physiological problems involved in handling and working with unknown material.

The technical program of the Symposium was opened by L. Costrell of the National Bureau of Standards who chaired the first session, which was devoted to scintillators. This session was highlighted by an invited paper presented by D. G. Thomas of the Bell Telephone Laboratories on the "Use of Semiconductors Doped with Isoelectronic Traps as Scintillation Counter Phosphors." A whole new class of doped materials has been found that might offer the possibility of higher efficiency scintillation detectors than present-day semiconductor detectors. A postdeadline paper presented by Nobel Prize winner R. Hofstadter of Stanford University dealt with the use of sodium iodide (thallium activated) scintillators for very high energy gamma-ray spectrometry. Dr. Hofstadter showed resolutions of about 1.3 percent for 10-GeV gamma rays. His

pioneer work in sodium iodide scintillators, first reported at the 1948 symposium, has resulted in the worldwide utilization of this scintillator in radiation detection.

The second session, chaired by R. T. Graveson of the AEC Health and Safety Laboratories, concentrated on tubes and other detectors. A paper by R. E. Simon of RCA explored the field of secondary electron emission and found that the application of certain solid-state concepts makes possible the development of a new type of secondary emitter. This emitter might be useful in producing multipliers with fewer stages, better statistics, and higher speeds than those now available.

Session three, chaired by I. L. Fowler of Atomic Energy of Canada Limited, was concerned with the increasingly popular and useful semiconductor detectors. This session opened with a description of "Studies of Detection Efficiencies and Operating Characteristics of Germanium-Lithium Detectors" by J. E. Cline of the National Reactor Testing Station, Idaho Falls. K. R. Zanio of the Hughes Research Laboratories discussed the transient response of cadmium telluride detectors to alpha particles. The alpha particles are controlled by trapping and detrapping—effects perhaps related to temperature. A more generalized presentation discussed the possibility of using cadmium telluride for the production of radiation detectors. This paper was presented by N. B. Strokan (for E. N. Arkad'eva) of the Ioffe Physical Technical Institute and was the first paper from the Soviet Union to be given at this series of symposia since its inception.

The fourth session, also devoted to semiconductors, was chaired by G. L. Miller of the Bell Telephone Laboratories. D. W. Aitken of Stanford University presented a comprehensive paper dealing with recent advances in x-ray technology. This was truly an international session since it included papers from Canada, France, the Soviet Union, and the United States. S. Chappell of the National Bureau of Standards presented a paper describing the pulse height response of silicon transmission detectors using thin films. N. B. Strokan of the U.S.S.R. presented two papers dealing with semiconductor counter resolutions and compensations of germanium radiation defects in the production of such counters.

The next session dealt with developments in spectrometry and circuits relating to detectors. This session was chaired by R. L. Chase of the Brookhaven National Laboratory and featured a paper by V. Radeka of the Brookhaven National Laboratory on pulse shaping. Problems of electrical noise, timing, and resolution were discussed for various applications with both types of detectors.

The final session, chaired by A. Roberts of the National

Accelerator Laboratories, presented papers on the combined topics of image tubes, spark chambers, and space applications. A number of the papers were concerned with spark chambers and spark chamber readout. Two of the papers involved image intensifiers, including one by G. T. Reynolds of Princeton University that described studies of weak light sources in biological systems.

The 58 papers presented at the Symposium, and an additional 19 submitted but not presented orally, will be published in the June issue of the IEEE Transactions on Nuclear Science. Copies will be available at a cost of \$10 each from the Institute of Electrical and Electronics Engineers, 345 E. 47th St., New York, N.Y. 10017.

HANDBOOK ON NEUTRON RADIATION PROTECTION

Because of their physical properties and biological effects, neutrons present a special type of radiation hazard. Moreover, the formulation of adequate protection regulations is extremely difficult because of the variable output of many neutron sources. NBS Handbook 63, *Protection Against Neutron Radiation Up to 30 Million Electron Volts*¹ (40 cents), originally published in 1957 and now reissued with corrections, reflects the thinking of the National Council on Radiation Protection on the recommended limits for maximum permissible dose of ionizing radiations.

In view of the rapid development of neutron technology, it was felt necessary to state recommendations rather than rules in many instances. However, in the section on protection against neutron radiation, rules are given which are deemed essential for proper protection. Other chapters cover the present status of physical and biological information, and radiation protection in installation and operation of neutron sources. A list of further references in this important field is appended.

BIBLIOGRAPHY OF SECOND BREAKDOWN IN SEMICONDUCTOR DEVICES

The use of transistors and other semiconductor devices is often limited by a phenomenon commonly called "second breakdown," a serious failure mode, especially in high-power, high-frequency transistors. The phenomenon is still not completely understood, but it is initiated by a spontaneous and usually abrupt reduction in the device voltage and a simultaneous internal constriction of current.

With the increase of transistor power and frequency capabilities, the problem of second breakdown has grown. It has become critically important in the design, fabrication, testing, and application of a wide range of transistor types.

The recent proliferation of papers on the subject makes a bibliography necessary to assist those concerned with the problem—people who work in fields ranging from consumer products to military equipment. NBS Technical Note 131, *Second Breakdown in Semiconductor Devices—A Bibliography*¹ (30 cents), answers that need.

Almost 200 references with appropriate key words are listed, providing a comprehensive coverage of the literature of second breakdown in transistors and other semiconductor devices from 1958 through 1967. An author index and a subject index are included, together with reference tabulations and key word assignments. A representative list of earlier papers dealing with what appears to be second breakdown in point-contact and p-n junction diodes is also included.

SCHEDULED NBS-SPONSORED CONFERENCES

Each year NBS sponsors a number of conferences covering a broad range of topics in science and technology. The conferences listed below are either sponsored or cosponsored by NBS and will be held at the Bureau's Gaithersburg, Md., facility unless otherwise indicated. These conferences are open to all interested persons unless specifically noted. For further information, address the person indicated below in care of Special Activities Section, Rm. A600, Administration Bldg., National Bureau of Standards, Washington, D.C. 20234.

Conference on Application of Newer Physical Techniques to the Study of Drug Metabolism. June 12–14. Cosponsors: Drug Research Board (NAS–NRC); National Institute of General Medical Sciences (NIH); Pharmaceutical Manufacturers Assoc. Foundation, Inc. Contact: D. C. Trexler (NAS–NRC).

Conference on the Structural Properties of Hydroxyapatite and Related Compounds. Sept. 11–13. Contact: W. E. Brown (NBS Polymers Division).

Measurements Technology. Sept. 17–18. Cosponsor: Scientific Apparatus Makers Association. Contact: G. E. Lawrence (SAMA).

Conference on Thermal Expansion. Sept. 18–20. Cosponsor: Westinghouse Astronuclear Laboratory. Contact: R. K. Kirby (NBS Metrology Division).

Performance of Buildings—Concept and Measurement. Sept. 23–25. Contact: W. W. Walton (NBS Building Research Division).

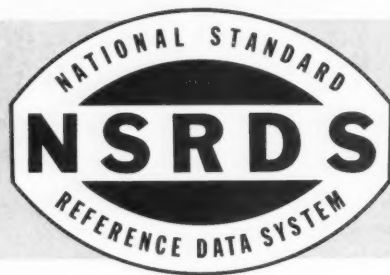
1968 International Conference on Modern Trends in Activation Analysis. Oct. 7–11. Cosponsors: U.S. AEC; International Atomic Energy Agency; EURISOTOP. Contact: P. D. LaFleur (NBS Analytical Chemistry Division).

Standards for High Pressure Research. Oct. 14–18. Contact: C. W. Beckett (NBS Heat Division).

Seminar on Durability of Insulating Glass. Nov. 14–15. Cosponsor: ASTM Committee E–6 on Methods of Testing and Building Construction. Contact: Henry Robinson (NBS Building Research Division).

Workshop on Mass Spectrometry. Nov. 18–19. Contact: A. J. Ahearn (NBS Analytical Chemistry Division).

¹ Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for the price indicated.



NEWS

This column regularly reports significant developments in the program of the National Standard Reference Data Systems. The NSRDS was established in 1963 by the President's Office of Science and Technology to make critically evaluated data in the physical sciences available to science and technology on a national basis. The System is administered and coordinated by the National Bureau of Standards through the NBS Office of Standard Reference Data, located in the Administration Building at the NBS Gaithersburg Laboratories.

What Are "Standard" Reference Data?

The mission of the National Standard Reference Data System is to provide critically evaluated data on the properties of substances to the U.S. technical community. But what does "critically evaluated" mean? Probably no two persons or projects would follow the same procedures for evaluation. In those projects associated with the National Standard Reference Data System, the "critical evaluation" varies widely. In some data centers, the experimental technique is reviewed, calculations are spot-checked, values of the fundamental constants are checked to ensure that the latest values were used, the temperature scale and other bases of measurement are checked (where appropriate), and limits of experimental uncertainty are independently assessed. In other centers, the data evaluator may decide, for intangible reasons that he may find difficult to formulate, that one particular value in the literature is "better" than another value. Such a judgment by a specialist of broad experience should not be underrated; the value obtained is more likely to be accurate than the result of unweighted averaging.

Most people agree that the first procedure provides a better "critical evaluation" than the second. However, for the practical purposes to which many compilations are applied, such a review is not justified, and the second procedure, or an intermediate one, is employed. The question then arises: What degree of critical evaluation is required for a compilation to be considered "standard" reference data? As the word "standard" has connotations that apply to few existing compilations, it is probably desirable to use "standard" sparingly. For the present, when meas-

urement results for most properties are uncertain and many are in dispute, the term "reference data" would avoid the implications aroused by the word "standard."

"Standard reference data," the ultimate goal of NSRDS, are to be striven for constantly, but perhaps not reached in many fields for years. Because of the variation in procedures for critical evaluation, it is intended that all future publications of NSRDS contain a statement of the criteria used for judgment and the argumentation used to derive the recommended values. Consideration is being given to publishing some compilations that would be designated "Reference Data" while others would receive the special designation "Standard Reference Data." At present, however, the question of where to draw the line between the two classes remains unanswered.

NMR Data Compilation

A new cooperative program associated with the National Standard Reference Data System, the National Bureau of Standards, and the National Research Council Office of Critical Tables has been inaugurated. This effort is a long-term program for data compilation and related information services in NMR spectroscopy of interest to chemists. Needs for quality criteria, present data resources, user requirements, format of data presentation, and instrument developments have been reviewed by an advisory panel under the auspices of the National Research Council Office of Critical Tables.¹

Currently, the program plans call for:

1. Intensive scrutiny and interlaboratory comparisons of a small group of selected molecules, with the purpose of determining the currently existing limits of precision and accuracy of NMR frequency measurements and related parameters.
2. Continuing compilation of tables of carefully evaluated, high precision chemical shifts and coupling constants based on retrospective and new data and including appropriate references. This compilation will stress quality before comprehensiveness. Major emphasis will be on proton NMR, but other nuclei will definitely be included.
3. A coordinating body that can act as a focus of attention on new developments, and as a nucleus for co-

operative efforts among all bodies concerned with NMR spectroscopy.

Present efforts under this program are on a volunteer basis. At a later date, some funding may be available to support some phases of the work. Anyone who is interested in participating, or in following the progress of the program, should communicate with any one of the people named below.

Dr. B. L. Shapiro,* Chairman
Ad hoc Subpanel on NMR Spectroscopy
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Pittsburgh, Pa. 15213

Dr. Thomas C. Farrar
Inorganic Materials Division
National Bureau of Standards
Washington, D.C. 20234

Dr. S. A. Rossmassler
Office of Standard Reference Data
National Bureau of Standards
Washington, D.C. 20234

Part 3 of Tables of Molecular Vibrational Frequencies

A compilation of vibrational frequency data for selected molecules is being made as a part of an international effort to compile and evaluate physical and chemical data. This activity is underway at the University of Tokyo in cooperation with the National Standard Reference Data Program. The third report in this effort is NSRDS-NBS-17, *Tables of Molecular Vibrational Frequencies, Part 3*² (30 cents).

(*Permanent address, effective Sept. 1, 1968: Department of Chemistry, Illinois Institute of Technology, Chicago, Ill. 60616)

It is a continuation of Part 1 published as NSRDS-NBS-6² (40 cents), and Part 2 published as NSRDS-NBS-11² (30 cents). Part 3 contains fundamental vibrational frequencies of 58 molecules together with vibrational assignments, sources of data, brief comments, and citations of references. The procedures used for the preparation of tables are the same as given in Part 1.

The fundamental frequencies are obtained mainly from the infrared and Raman spectra. When these are not available, other experimental data, such as microwave results, are taken into account. The selection of vibrational fundamentals from observed data is based upon careful studies of the spectral data and comprehensive mathematical analyses.

The tables were designed to provide a concise summary needed for the computation of ideal gas thermodynamic properties. They may also provide a convenient source of information to those who require vibrational energy levels and related properties in molecular spectroscopy, analytical chemistry, and other fields of physics and chemistry.

Three Recent Publications of the Atomic and Molecular Processes Information Center

The Atomic and Molecular Processes Information Center, located at Oak Ridge National Laboratory and under the sponsorship of the U.S. Atomic Energy Commission and the Office of Standard Reference Data, serves the technical community by collecting, storing, evaluating, and disseminating atomic and molecular processes information.

ORNL-AMPIC-5, *International Directory of Workers in the Field of Atomic and Molecular Collisions*,³ is a worldwide list of scientists engaged in or interested in the field of electronic, atomic, and molecular collisions. It is a revision of the 1965 directory. For the revision, effort was made to contact university departments and other laboratories directly to determine who should be included. Many names, however, were derived from the open literature and from attendees at topical conferences. Since the directory is now computerized, corrections and additions

continued

are readily made. The Atomic and Molecular Processes Information Center requests that changes in addresses and additional appropriate names be brought to its attention. All changes will be incorporated in an annual revision. Correspondence on the directory should be addressed to:

J. A. Ray
Atomic and Molecular Processes Information Center
P. O. Box Y
Building 9201-2
Oak Ridge, Tenn. 37830

ORNL-AMPIC-8, *Bibliography of Atomic and Molecular Processes for July-December 1966*,³ and ORNL-AMPIC-9, *Bibliography of Atomic and Molecular Processes for January-June 1967*,³ are annotated bibliographies containing references of interest to atomic and molecular processes research. Bibliographic sources consisted of seventy-six scientific journals and five abstract journals. The references are classified into fourteen major categories with appropriate subcategories. Each entry in the respective category is entered alphabetically and includes the reactants or the atomic and molecular system of interest.

Bibliography of Evaluation of Information Systems

Of interest to operators of and workers in information and data centers is the recent publication, NBS Technical Note 297, *Evaluation of Information Systems: A Selected Bibliography With Informative Abstracts*² (\$1). The author is Madeline M. Henderson. This annotated bibliography covers material published or available through June 1965.

The subject matter covered is the evaluation of information retrieval systems or document reference systems. The scope does not extend to data processing systems, nor does it include the equipment used in such systems—the hardware per se. Equipment is considered only as a component of a total system that is being evaluated. The emphasis is on evaluation or testing, rather than design or selection of systems. There is definite overlap in the criteria applied in design and those used in evaluation, but the orientation of the work reported has been the guiding factor in selecting material for inclusion in this bibliography. The evaluations may be of total systems, or of components or subsystems of total systems. Such systems may be in operation, or may be experimental and set up specifically for a testing program. The purpose of the abstracts is to give a summary of the corresponding paper, report, or book section. The abstracts were prepared so as to be informative; the author's own wording has been used extensively, in order to avoid misinterpretation. The citations and abstracts in this bibliography are grouped in four categories, according to their primary purpose or scope: comparative evaluation, descriptive evaluation, discussion of evaluation factors, and proposals. Those inter-

ested in such systems will find this bibliography an extensive compilation of valuable material on the subject of evaluation of information systems.

More Efficient Use of Teletypewriter as a Remote Computer Console

Another recent publication which data center operators will find useful is NBS Technical Note 419, *Accessory Equipment and Teletypewriter Modifications for Remote Computer Consoles*² (20 cents), by Charles H. Popenoe and Robert C. Thompson. It explains how equipment auxiliary to and simple modifications of a teletypewriter enable it to be used more efficiently as a remote console of a computer.

A number of laboratories in the NBS Heat Division have made considerable use in recent years of punched-paper tape for data recording and computer processing. This program has yielded much first-hand experience in the use of remote-access, time-shared computing systems, first at Dartmouth College, then at several commercial installations. These facilities have in common a simplified, convenient, user-oriented language called BASIC,⁴ and remote-access input-output terminals provided by a teletypewriter operated over commercial telephone lines.

Experience gained during this use motivated the authors to make a number of modifications and improvements and to design auxiliary devices to increase the efficiency of the terminal when used in routine data editing or as computer consoles. In most cases, a simple mechanical or electrical modification to the machine has sufficed to provide increased facility in its use as a remote terminal. It has been found that considerable savings in time may be realized by the use of a word generator, which provides for the automatic typing of often used programming instructions and system commands.

This type of accessory equipment is especially useful for the nontypist, speeding the typing, and eliminating typing errors. In the BASIC computer language, it is necessary to number each line in increasing order. To aid in this task, a sequential numbering generator was constructed and found to be a real time saver.

This report documents the accessory equipment and modifications found to be necessary or desirable in using a teletypewriter as a remote computer console. Among the items treated are pushbutton word generators, providing automatic typing of often used programming instructions. Also covered are option switches for full- or half-duplex operation, for inserting the sixth bit for lower-case letters and the eighth bit for parity compatibility.

¹See, NMR spectroscopy panel meeting, NSRDS News, NBS Tech. News Bull. 52, No. 2, 37 (Feb. 1968).

²Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for the price indicated.

³Available from the Atomic and Molecular Processes Information Center, Oak Ridge National Laboratory, P.O. Box Y, Oak Ridge, Tenn. 37830.

⁴A Manual for BASIC, by J. G. Kemeny and T. E. Kurtz, Dartmouth College, Dartmouth Computation Center, Dartmouth Bookstore, Hanover, N.H. (1965).

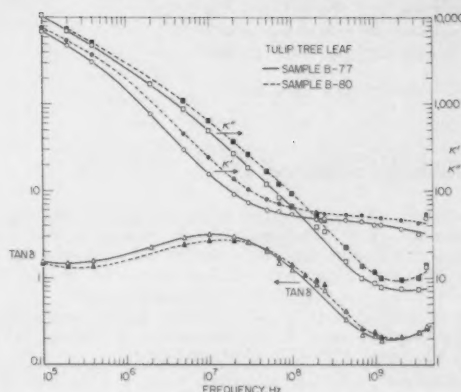
DIELECTRIC CONSTANT *continued*

tulip tree, philodendron, passiflora, dandelion, buckhorn, dogwood, and sugar maple.

The results showed remarkably similar values for all leaves studied. This was interpreted as an indication that the predominant effect of the dielectric properties of leaves is the interfacial polarization resulting from the buildup of ions (present in the cell sap) at the plant cell walls.

In addition to the study of leaves, measurements were made on specimens prepared from tulip tree twigs. Measurements of clay soil samples with varying amounts of moisture content were also included in the study.

Plots of the real and imaginary parts of the relative dielectric permittivity and the loss factor of two samples (77 and 80 percent moisture by weight) of tulip tree leaves as a function of frequency at 23°C.



PUBLICATIONS of the National Bureau of Standards*

PERIODICALS

Technical News Bulletin, Volume 52, No. 4, April 1968, 15 cents. Annual subscription: Domestic, \$1.50; foreign, \$2.25. Available on a 1-, 2-, or 3-year subscription basis.

Journal of Research of the National Bureau of Standards

Section A. Physics and Chemistry. Issued six times a year. Annual subscription: Domestic, \$5; foreign, \$6. Single copy, \$1.

Section B. Mathematical Sciences. Issued quarterly. Annual subscription: Domestic, \$2.25; foreign, \$2.75. Single copy, 75 cents.

Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic, \$2.75; foreign, \$3.50. Single copy, 75 cents.

CURRENT ISSUES OF THE JOURNAL OF RESEARCH

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Creep rupture properties of Ti-8Al-1Mo-1V alloy. W. D. Jenkins and W. A. Willard.

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continued
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*Publications for which a price is indicated are available by purchase from the Superintendent of Documents, U. S. Government Printing Office, Washington, D.C. 20402 (foreign postage, one-fourth additional). The NBS nonperiodical series are also available from the Clearinghouse for Federal Scientific and Technical Information, Springfield, Va. 22151. Reprints from outside journals and the NBS Journal of Research may often be obtained directly from the authors.

CLEARINGHOUSE BIBLIOGRAPHIC JOURNALS**

U.S. Government Research & Development Reports. Semimonthly journal of abstracts of R&D reports on U.S. Government-sponsored projects and U.S. Government-sponsored translations of foreign technical material. Annual subscription (24 issues): Domestic, \$30; foreign, \$37.50. Single copy, \$3.

U.S. Government Research & Development Reports Index (formerly *Government-Wide Index to Federal Research & Development Reports*). Semimonthly index to preceding; arranged by subject, personal author, corporate author, contract number, and accession/report number. Annual subscription (24 issues): Domestic, \$22; foreign, \$27.50. Single copy, \$3.

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